

Analytical Chemistry Laboratory

Highlights for July through September 2000

The Analytical Chemistry Laboratory (ACL) provides analytical chemistry services and technical support for a variety of programs at ANL, works collaboratively with programs, and conducts independent research in analytical chemistry. For more information on any of these highlights, call one of the individuals listed with the highlight or the ACL Office at 630-252-4473.

Information about the ACL is available through its World Wide Web home page at <http://www.cmt.anl.gov/acl/acl.htm>. Topics available from this home page include:

- ✓ Advice on how to submit samples
- ✓ Major technical specialties of the ACL staff
- ✓ ACL Quarterly Highlights since October 1994
- ✓ ACL Annual Reports for FY 1996 through 1999
- ✓ List of all published ANL/ACL technical reports

David W. Green, Manager

Technical Support for National Analytical Management Program (W. E. Streets)

The ACL is supporting the National Analytical Management Program (NAMP) in the development of centralized information systems that will allow DOE complex personnel to share pertinent information to promote quality initiatives administered by the Office of Environmental Management (EM). The NAMP Laboratory Informational Network (NAMPnet) provides a single web-based point for users to input and access information about EM contacts at the various sites, costs of analyses, laboratory contracts, audits, and participation in, and assessment of, performance in national performance evaluation programs. Much of the work with databases is done with the assistance of the CMT Computer Applications, Network, and Security Group (J. Copple, T. Scandora, J. Kulaga, and R. Krol).

Twenty NAMP users requested and received passwords to the information in the restricted section of the NAMPnet web site. New contact information was added to the common administrative database. We have collected additional results data from the national performance evaluation programs currently used in the Integrated Performance Evaluation Program and loaded these into the performance evaluation results database.

The ACL provides technical and advisory support to NAMP for other tasks as requested. We completed review of the Multi-Agency Radiation Laboratory Protocols Manual. We are advising the interagency Methods and Data Comparability Board on the design and development of the National Environmental Methods Index, a web-based database.

Final Method for High-Precision Measurement of Lithium and Aluminum by ICP/AES (D. G. Graczyk, S. J. Lopykinski, and A. M. Essling)

Over the past year and a half, ACL analysts have pursued a practical implementation of methodology adapted from work done at the National Institute of Standards and Technology (NIST) to achieve highly precise measurements of lithium and aluminum concentrations in lithium aluminate ceramics with the inductively coupled plasma-atomic emission spectrometry (ICP/AES) technique. In the process, we discovered that results obtained from the method were more dependent on instrument operating conditions and lithium isotopic composition than the NIST experience divulged. By repeatedly challenging the process and finding ways to understand and resolve difficulties that showed themselves, we discovered ways to improve and standardize the operations. This past quarter, these efforts culminated in a set of practical operating procedures that incorporate all of the insights we gained to control matrix effects, isotope effects, and instrument operations. We also worked with colleagues at Pacific Northwest National Laboratory to modify a software tool they had developed for data reduction to include automated application of formulas we derived to make corrections for isotopic differences in lithium among samples and standards. The combination of our detailed operating instructions and this modified software tool provides a methodology that is relatively easy to apply and that, we believe, will be readily transferable to any capable laboratory. It is especially noteworthy that, although the method was established for lithium and aluminum assays in lithium aluminate ceramics, its implementation was purposely formulated in general terms so that it should adapt almost effortlessly to other elements and applications.

A measure of the level of performance available from the methodology was obtained from a test series completed this quarter. In these tests, a set of five simulated samples containing known amounts of lithium

and aluminum was prepared by careful gravimetric mixing of stock solutions that included lithium enriched in the ^6Li isotope. The simulated samples contained lithium with natural isotopic composition (7.6 wt% ^6Li), 20 wt% ^6Li , 30 wt% ^6Li , 40 wt% ^6Li , and 95 wt% ^6Li . These five samples were analyzed nine separate times, with the torch assembly in the ICP system disassembled and replaced every third run. The pooled relative standard deviation of the nine runs of five samples was 0.06% for lithium and 0.02% for aluminum. Compared to the as-made concentrations of the simulated samples, lithium showed an average relative difference of -0.05% and aluminum of -0.06%. The procedures seem to work very well indeed.

Lithium Aluminate Methods Surveillance (D. G. Graczyk, A. M. Essling, S. J. Lopykinski, F. P. Smith, and F. J. Martino)

Methods developed in the ACL for chemical analysis of lithium aluminate ceramics to determine major constituents and impurities were evaluated in an on-site surveillance during the week of September 18 through 22. Two representatives of the sponsoring program at Pacific Northwest National Laboratory observed ACL analysts as they carried out operations according to Standard Operating Procedures (SOPs) that implement ACL methods for preparing samples and making instrumental measurements to provide the desired results. Sample materials processed in the exercise were selected to test the analytical performance of each procedure relative to specific program requirements. Operations that were demonstrated for evaluation included potassium hydroxide fusion and ion chromatography to determine halide impurities. Also demonstrated was microwave-assisted acid dissolution of the ceramic, followed by high-precision ICP/AES measurement of lithium and aluminum. Finally, measurement of 24 cationic impurities by conventional ICP/AES was demonstrated. The data from each set of measurements were evaluated in terms of sensitivity (detection limits), precision, and potential bias.

Operations during the surveillance went quite smoothly, and performance of every method was deemed acceptable from assessment of the generated results. The success of this surveillance opens the door for ACL's transfer of the procedures to a commercial laboratory under contract to the program, and for ACL's use of the procedures in preparing and characterizing working reference materials for the program. Work to accomplish these objectives is underway.

Publications

"Balance is the Key," D. W. Green, *Managing the Modern Laboratory* 4(4), 56A-58A (2000).

Talks Presented

"National Analytical Management Program (NAMP) Projects at Argonne National Laboratory," W. E. Streets, CMT Technical Seminar Series, July 7, 2000.

"Role of Analytical Laboratories within EM," D. W. Green, presented at the 18th Meeting of the DOE Analytical Managers Group, Denver, CO, August 14-17, 2000.

"Have Sample Will Travel—Analysis of Toxic Substances," A. S. Boparai, presented at Illinois Institute of Technology, Chicago, IL, September 13, 2000.

"Practical High Precision Assays by Inductively Coupled Plasma Atomic Emission Spectrometry," D. G. Graczyk, CMT Technical Seminar Series, September 29, 2000.

University of Chicago Review of the Chemical Technology Division, Argonne National Laboratory, Argonne, IL, September, 2000:

"Analytical Chemistry Laboratory Work-for-Others (WFO) Programs," F. J. Martino, poster session; "National Analytical Management Program," W. Elane Streets and D. W. Green, poster session; "Modernizing Methods for Chemical Analysis of Lithium Aluminate Ceramics," D. G. Graczyk, presentation.

The "Request for Technical Services" Form (ANL-80) is available on-line through the ANL forms page: <http://www.ipd.anl.gov/aim/forms>

If you submit samples regularly, you can save a copy to avoid starting from scratch with this form for the next set of samples. The form can be sent electronically to the ACL analyst or group leader, but it should be received from the person who normally signs the "Cost Code Authorization" box on the hard copy version. There can be some advantage to sending a printed hard copy of the form with the samples to be analyzed.